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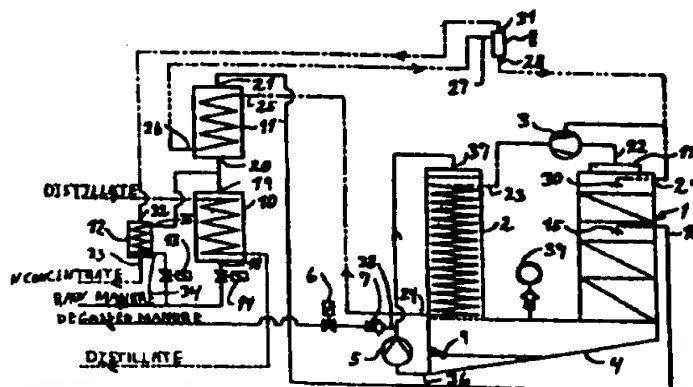
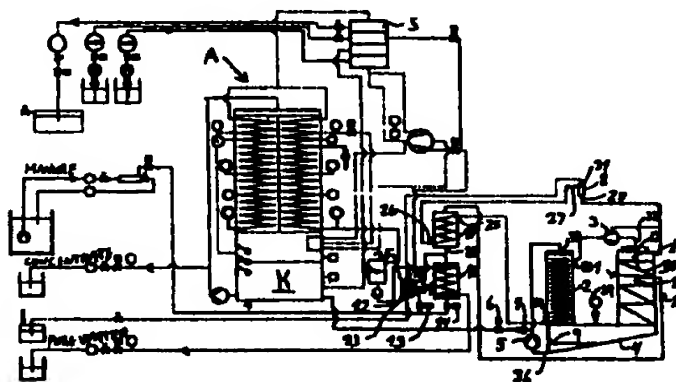
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(57) Abstract

The invention relates to a method of degassing liquid biological waste and an apparatus for use by the method. The invention is particularly suited for degassing manure of highly volatile components such as carbon dioxide, CO<sub>2</sub>, and ammonia, NH<sub>3</sub>. Degassing is performed using the natural components of the manure and involving a strongly reduced energy consumption. Degassing takes place by compressing the highly volatile components and using the highly volatile components as an energy transfer medium in heat exchangers to subject the manure to a temperature rise before the manure is degassed.



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**METHOD AND APPARATUS FOR SEPARATING VOLATILE COMPONENTS FROM A LIQUID.**

The present invention relates to a method of purifying a liquid that contains more volatile components and less volatile components during evaporation of mixed liquids, wherein the liquid is heated to the boiling point in a boiler, and wherein the vapours are purified of undesirable gaseous components in the form of the more volatile components, after which the purified gases are condensed by heat exchange with unpurified vapours, while the less volatile components are concentrated in the boiler.

The invention also relates to an apparatus for use by the method.

The present invention will now be explained in detail in the context of the problems associated with the purification of biological waste water, preferably in the form of manure, during an evaporation process. The evaporation process will preferably be a vapour compression process. However, the evaporation process may also take place without compression of vapours, which are formed in the boiler and which are subsequently conducted to the heat exchanger for condensation.

The method and the apparatus according to the invention may also be used for the purification of other liquids.

When evaporating liquid containing more volatile components and less volatile components, it is desirable for the amount of more volatile components to be as small as possible. This is due to the fact that the more volatile components often consist of gases that are not condensable at a pressure and temperature corresponding to the temperature and pressure for the evaporation of the basic liquid of the mixture, which is ordinarily water. The non-condensable components will impede the evaporation of water as they place themselves as an insulating layer in the heat exchanger and thus impede the heat transmission to the condensable components in the heat exchanger. This will cause an energy consumption increase.

Especially when evaporating biological waste containing carbon dioxide and ammonia as more volatile components and fatty acids and mineral salts as less volatile components

nents, it is desirable to reduce the contents of carbon dioxide and ammonia in the liquid.

On removal of carbon dioxide and ammonia from the liquid it can be better prepared for the purification taking place during evaporation, in which it is desired to separate as large amounts of pure water vapour as possible and to obtain the largest possible concentration in the boiler. Such a process may run batch-wise or more or less continuously either by a batch-wise or continuous exchange of the concentrated mixture in the boiler.

Thus, through the evaporation process it is desired to obtain as high a share of water vapour as possible since the non-condensable components, such as carbon dioxide, will take space from the water vapour as the non-condensable gases are carried along from the boiler to the heat exchanger. In a compression step, if any, this will lead to an increased energy consumption for the compression. Furthermore, as mentioned above, the heat transmission in the heat exchanger is impeded. For these reasons it is desirable to remove carbon dioxide before the evaporation is performed. It is also desirable to remove and bind carbon dioxide in order to prevent environmental pollution.

It has previously been proposed to separate ammonia before the vapours are let into the heat exchanger by taking them through a scrubber. Although this has proven effective, it involves disadvantages, however, since it requires the addition of various chemicals. This leads to an increase of the purification costs.

The ammonia contained in the biological waste is generally undesirable in the evaporation process due to pollution of the evaporated liquid. As ammonia will evaporate and may condense with the water vapour in the heat exchanger, the condensate will be polluted with ammonia. If the condensate is to be dischargeable directly to a recipient, it will be necessary to remove the ammonia in a different manner. This may take place by the addition of chemicals. This is necessary, but undesirable, since it requires further processing steps and will lead to heavy purification costs. It is desirable to reduce

these costs by reducing the content of ammonia in the mixed liquid in order at least to reduce the chemical consumption in the scrubber.

5 During purification it is possible to perform a concentration of the less volatile components, such as fatty acids and mineral salts, in the basic liquid that remains in the boiler. This concentrated amount can be discharged from the boiler.

10 A method and an apparatus of the known type may be designed according to the teachings of international patent application No. WO 92/03203. The liquids that may be purified by a method according to the present invention, in addition to biological waste water, which among other things comprises manure, may also comprise emulsified liquids, such as cooling/lubricating oil emulsions, degreasing water, oil containing wastewater, wastewater from laundries, solvents and wastewater from food production and the like.

15 The object of the present invention is to provide a method and an apparatus for purifying liquids, wherein it is possible to remove more volatile components before an evaporation takes place, and wherein a low energy consumption and little use of chemicals for separation of the volatile components is obtained as the more volatile and the less volatile components can be brought to react and form useful substances, and said method and said apparatus being particularly suited for use in the purification of biological wastewater.

20

25 This object is obtained according to the present invention by a method that is characterised in that the liquid is heated before it is degassed, that degassing takes place by conducting the liquid to a column where the more volatile and less volatile components are separated and from which column the more volatile components in the form of vapour are conducted to a primary side of a first heat exchanger so that the more volatile components are condensed and discharged from the primary side, and from  
30 which column the less volatile components in the form of liquid are conducted to a secondary side of said first heat exchanger where they are heat exchanged with the condensed more volatile components so that further fractions of more volatile compo-

nents are evaporated in the secondary side and conducted to said column, from where, in the form of vapour, they are conducted to the primary side of said first heat exchanger, and that after complete or partial separation of the more volatile components the heated liquid is conducted to the boiler.

5

An apparatus for use by the method and comprising a boiler and a heat exchanger is characterised in that the apparatus further comprises:

- a column provided with an inlet for a liquid containing more volatile and less volatile components, a discharge for more volatile components, and an outlet for less volatile components,
- a first heat exchanger provided with an inlet and an outlet of a primary side for the more volatile components and an inlet and an outlet of a secondary side for the less volatile components,
- a separator with an inlet, which is connected with the outlet from the primary side for the more volatile components.

15

The method according to the invention is advantageous in that the more volatile components are separated from the liquid at a very low energy consumption. Some of the energy for heating the liquid is taken from the more volatile components after the more volatile components have been conducted through the outlet from the first heat exchanger. Initially the more volatile components are used for heating the less volatile components after the more volatile components have been conducted through the outlet from the column and subsequently compressed.

20

- 25 When conducting the compressed more volatile components through the primary side of the heat exchanger and the less volatile components through the secondary side of the heat exchanger, the compressed more volatile components will contribute to a heating of the less volatile components. This liquid, which contains less volatile components, may contain a further share of the more volatile components. Through several passages through the column and the heat exchanger the less volatile components will
- 30 constantly be subjected to energy addition. In this manner the further share of the

more volatile components will evaporate and be carried along to the column, from where they circulate to the primary side, where they condense.

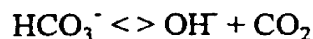
5 During the energy transfer from the compressed more volatile components to the less volatile components a partial condensation of the more volatile components takes place and it becomes possible to separate a very large share of the more volatile components from the liquid mixture.

10 The more volatile components will often partly condense in the heat exchanger. This liquid/gas mixture is conducted to the separator. In this manner the more volatile components are separated into a gaseous fraction and a liquid fraction. The liquid fraction is conducted back to the column for repeated treatment as discussed above (reflux). The gaseous fraction condenses and is conducted back to the container for use, e.g. as concentrated fertiliser. The gaseous fraction may be conducted via a third  
15 heat exchanger, where a condensation takes place during heating of the incoming liquid mixture.

Particularly in the context of evaporation of liquid biological waste, e.g. manure, containing among other things carbon dioxide,  $\text{CO}_2$ , and ammonia,  $\text{NH}_3$ , as more volatile  
20 components it is necessary to conduct the more volatile components to a third heat exchanger for condensation. During condensation the carbon dioxide is brought to react with the ammonia for the formation of ammonium hydrogen carbonate,  $\text{NH}_4\text{HCO}_3$ . The ammonium hydrogen carbonate may be stored in an ordinary closed container and, therefore, it is subsequently conducted to a container for use as a fertiliser component.  
25

The liquid biological waste, e.g. in the form of manure, must be given a temperature corresponding to the boiling point of the liquid biological waste. For manure containing carbon dioxide and ammonia it is necessary for the temperature of the manure to  
30 be above  $80^\circ\text{C}$  as carbon dioxide is almost insoluble in a liquid at temperatures above  $80^\circ\text{C}$ . When the temperature in the column is above the boiling point e.g. of the ma-

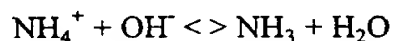
nure, the carbon dioxide cannot perform an ionic bond. This causes the carbon dioxide to exit from the liquid at the following equilibrium:



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It is seen that this equilibrium results in an alkaline reaction. This alkaline reaction causes the ammonium to be more volatile and likewise exit from the liquid at the following equilibrium

10



By the method and the apparatus according to the invention it is possible in liquid biologic waste to remove up to at least 99% of the carbon dioxide and up to at least 75% of the ammonia. This has been achieved in experiments with manure having an ammonium content of 5000 mg/l.

15

As the ammonia content is reduced considerably, there will be a substantial saving in the chemical consumption for removal of the remaining ammonia content, e.g. in a scrubber. In this manner a cost saving is achieved in the purification.

20

In a preferred embodiment the apparatus is characterised in that it further comprises a compressor, which is arranged between the outlet in the column for the more volatile components and the inlet of the first heat exchanger for the more volatile components, and that it further comprises

25

- a second heat exchanger having an inlet to a primary side from the outlet of the primary side of the first heat exchanger, an outlet from the primary side, an inlet to a secondary side for the liquid containing the more volatile components and the less volatile components, and an outlet from the secondary side for the liquid containing the more volatile components and the less volatile components, wherein the outlet from the primary side is in connection with the inlet to the separator, and

30



- a third heat exchanger with an inlet to a primary side for a gaseous fraction of the more volatile components from an outlet from the separator and an outlet from the primary side for a condensate of the more volatile components.

5 The invention will now be explained in detail with reference to the accompanying drawing, wherein

Fig. 1 shows a plant for waste water processing and comprising an apparatus according to the invention,

10 Fig. 2 shows a partial view of part of the plant shown in Fig. 1, and

Fig. 3 shows a partial view corresponding to Fig. 2 but of a second embodiment of the apparatus according to the invention.

15 Figure 2 of the drawing constitutes a part of Fig. 1. Both figures will be explained simultaneously. Fig. 1 shows a plant designed to handle biological wastewater in the form of manure. In Fig. 1 the unit designated with an A is a plant of the type described in international patent application WO 92/03203, the contents of which are hereby incorporated by reference.

20 In principle, the apparatus according to the invention may be said to constitute one unit, wherein a degassing is performed on the manure present in the boiler K of the apparatus A. Hereby it is possible to reduce the chemical consumption in the scrubber S of the plant.

25 The functioning of the main plant will not be described in detail. The description will focus on the part of the plant shown in Figs. 2 and 3, which show a first and a second embodiment, respectively, of an apparatus according to the invention.

30 Fig. 1 is a schematic view of an embodiment of an apparatus according to the invention. The apparatus comprises a column 1, a first heat exchanger 2, a compressor 3, a vessel 4 for the column 1, a circulation pump 5, a motor valve 6, a check valve 7, a gas/liquid separator 8, a level sensor 9 for control of the liquid level in the vessel 4, a

first section 10 of a second heat exchanger, a second section 11 of the second heat exchanger, a third heat exchanger 12, a first motor valve 13 and a second motor valve 14. The column 1 is provided with nozzles 15 for discharge of liquid biological waste in the column 1.

5

The column 1 is provided with an inlet 16 for the liquid biological waste. As an example of liquid biological waste the following part of the description will use manure. The manure is conducted to the column 1 to be separated in the column 1 into more volatile components and less volatile components. The more volatile components are conducted to a foam inhibitor 17 in the upper part of the column 1. The less volatile components are conducted to the vessel 4 in the bottom of the column 1. When the liquid biological waste is manure, the more volatile components comprise among other things carbon dioxide,  $\text{CO}_2$ , and ammonia,  $\text{NH}_3$ , and the less volatile components comprise among other things water ( $\text{H}_2\text{O}$ ), fatty acids and mineral salts.

10

Before the manure is conducted to the column 1, the manure is heated as it is conducted through a secondary side of the first section 10 and a secondary side of the second section 11 of the second heat exchanger. The first section 10 of the second heat exchanger is provided with an inlet 18 for the manure. The inlet 18 is provided with the motor valve 14 for inlet of manure to the first section 10. The first section 10 is provided with an outlet 19 leading to an inlet 20 to the second section 11 of the second heat exchanger. The second section 11 is provided with an outlet 21 leading to the inlet 16 to the column 1. The manure is to obtain a temperature rise to the boiling point of the manure before the manure is conducted to the nozzles 15 in the column 1.

15

After the manure has been conducted through the nozzles 15 in the column 1 and, as mentioned, has been separated into more volatile components and less volatile components, the more volatile components are conducted from an outlet 22 from the foam inhibitor 17 to the compressor 3, where the more volatile components are compressed.

20

After the more volatile components have been compressed, the more volatile components are conducted to an inlet 23 of a primary side of the first heat exchanger 2. The more volatile components are conducted through the primary side of the first heat ex-

changer 2 and to an outlet 24 of the primary side of the first heat exchanger 2. The more volatile components are then conducted to an inlet 25 of a primary side of the second section 11 of the second heat exchanger. The more volatile components are conducted through the primary side of the second section 11 and to an outlet 26 of the  
5 second section 11.

After having been conducted through the primary side of the first heat exchanger and the second section 11 of the second heat exchanger, respectively, the more volatile components have partly condensed and consist of a gaseous fraction and a liquid frac-  
10 tion. From the outlet 26 of the primary side of the second section of the second heat exchanger the gaseous fraction and the liquid fraction of the more volatile components are conducted to an inlet 27 to the gas/liquid separator 8.

The liquid fraction is conducted from an outlet 28 of the separator 8 to an inlet 29 for the liquid fraction and to nozzles 30 in the column 1 as reflux. The gaseous fraction of the more volatile components is conducted from an outlet 31 to an inlet 32 to a pri-  
15 mary side of the third heat exchanger 12. In the third heat exchanger 12 a complete condensation of the gaseous fraction takes place. During condensation of the gaseous fraction carbon dioxide,  $\text{CO}_2$ , diffuses into the liquid and together with water,  $\text{H}_2\text{O}$ , and ammonia,  $\text{HN}_3$ , ammonium hydrogen carbonate,  $\text{NH}_4\text{HCO}_3$ , is formed. The am-  
20 monium hydrogen carbonate is conducted to an outlet 33 from the primary side of the third heat exchanger and may be stored in an ordinary closed container (not shown) and discharged from there.

25 Parts of the manure that are conducted to the second heat exchanger are initially conducted to a secondary side of the third heat exchanger 12. The manure is conducted to an inlet 34 of the secondary side of the third heat exchanger 12 by means of the motor valve 13 to an outlet 35 from the secondary side. From the outlet 35 of the secondary side of the third heat exchanger 12 the manure is conducted to the inlet 20 of the sec-  
30 ondary side of the second section 11 of the second heat exchanger.

The less volatile components are conducted, as mentioned, to the vessel 4. From the outlet 36 from the vessel the less volatile components are conducted in liquid form by means of a circulation pump 5 to an inlet 37 of a secondary side of the first heat exchanger 2. The level sensor 9 in the vessel 4 ensures that the less volatile components are not conducted from the vessel 4 until a sufficient amount of the less volatile components is present in the vessel 4. The less volatile components are conducted through the secondary side of the first heat exchanger 2, for evaporation of any remaining more volatile components, and back to the vessel 4. At intervals, during the circulation of the less volatile components from the vessel 4 and through the secondary side of the first heat exchanger 2, parts of the liquid containing the less volatile components will be discharged. Discharge takes place through an outlet 38, through the check valve 7 and the motor valve 6. The part of the liquid containing the less volatile components that is discharged may be described as degassed, i.e. free of more volatile components, and may be conducted on to further treatment, if any, such as evaporation.

During compression in the compressor 3 the more volatile components will reach a temperature that is higher than the temperature of the manure conducted to the nozzles 15 in the column 1.

An energy liberation takes place when the more volatile components are conducted through the primary side of the first heat exchanger 2, through the second section 11 of the second heat exchanger and after separation in the separator 8 through the primary side of the third heat exchanger 12. During energy liberation a partial or complete condensation of the less volatile components takes place.

An energy absorption takes place when the manure is conducted through the secondary side of the third heat exchanger 12 and through the secondary side of the second section 11 of the second heat exchanger. During energy absorption a temperature rise of the manure takes place.

En energy absorption takes place when the less volatile components are conducted through the secondary side of the first heat exchanger. During energy absorption a

temperature rise of the less volatile components takes place. If energy transfer from the more volatile components in the primary side of the first heat exchanger 2 to the less volatile components in the secondary side of the first heat exchanger 2 is not sufficient, it is possible by means of a secondary energy induction device 39 to induce a further temperature rise into the less volatile components.

The apparatus illustrated in Fig. 3 differs from the apparatus shown in Fig. 2 in that the heat exchangers 10, 11, 12 have been left out. Thus, in Fig. 3 the raw manure will be input directly into the column 1 without prior heat exchange in a heat exchanger corresponding to the heat exchangers 10, 11, 12. It should be noted, however, that the raw manure must have been subjected to a heating so that its temperature is in the vicinity of the boiling point before it is let into the column 1. Furthermore, the gas/liquid mixture discharged from the heat exchanger 2 will be conducted directly to the gas/liquid separator 8, from where a liquid fraction containing a concentrated amount of nitrogen compounds is discharged in the form of vapour and where a liquid fraction is conducted back to the column 1.

In the embodiments shown the apparatus is illustrated with a compressor 3. The compressor 3 is inserted to use a vapour compression principle in the apparatus shown in Figs. 2 and 3. However, the apparatus according to the invention may also be produced without the compressor 3. In such an embodiment the energy economy would be slightly poorer.

The invention has been described above with reference to two specific embodiments of an apparatus according to the invention. It will, however, be possible to exercise the method according to the invention with other embodiments of apparatuses and, likewise, other embodiments of the apparatus according to the invention may be provided. Also, other biological waste materials than manure may be treated by the method according to the invention and by means of the apparatus according to the invention.

## CLAIMS

1. A method of purifying a liquid that contains more volatile components and less volatile components during evaporation of mixed liquids, wherein the liquid is heated  
5 to the boiling point in a boiler (K), and wherein the vapours are purified of undesirable gaseous components in the form of the more volatile components, after which the purified gases are condensed by heat exchange with unpurified vapours, while the less volatile components are concentrated in the boiler, c h a r a c t e r i s e d in that the liquid is heated before it is degassed, that degassing takes place by conducting the  
10 liquid to a column where the more volatile and less volatile components are separated and from which column the more volatile components in the form of vapour are conducted to a primary side of a first heat exchanger so that the more volatile components are condensed and discharged from the primary side, and from which column the less volatile components in the form of liquid are conducted to a secondary side of said  
15 first heat exchanger where they are heat exchanged with the condensed more volatile components so that further fractions of more volatile components are evaporated in the secondary side and conducted to said column, from where, in the form of vapour, they are conducted to the primary side of said first heat exchanger, and that after complete or partial separation of the more volatile components the heated liquid is conducted to  
20 the boiler.

2. A method according to claim 1, c h a r a c t e r i s e d in that resulting components, in the form of condensate and remaining gases, from the discharge of the primary side are conducted to a gas/liquid separator, and that a liquid fraction from here is returned  
25 to the column while a gaseous fraction from here with a concentration of the more volatile components is collected for use.

3. A method according to claim 1 or 2, c h a r a c t e r i s e d in that the more volatile components are compressed before the heat exchange step.

30

4. A method according to claim 1, 2 or 3, characterised in that the more volatile components comprise carbon dioxide and ammonia, and that the less volatile components comprise water, fatty acids and mineral salts.
5. A method according to any one of the preceding claims, characterised in that the more volatile components are conducted from the primary side of the first heat exchanger to a primary side of a second heat exchanger and on to the separator, preferably from the first heat exchanger to a primary side of a first section of a second heat exchanger, on to a primary side of a second section of the second heat exchanger and on to the separator.
6. A method according to claim 4, characterised in that the gaseous fraction of the more volatile components is conducted from the separator to a primary side of a third heat exchanger, that the gaseous fraction is retained in the heat exchanger, that a temperature of the gaseous fraction is lowered to a temperature not exceeding the condensation temperature of ammonia, that the ammonia is condensed to a condensate, that carbon dioxide is diffused into the condensate, and that the carbon dioxide is made to react with the ammonia for formation of ammonium hydrogen carbonate.
7. A method according to any one of the preceding claims, characterised in that the liquid containing the more volatile components and the less volatile components is conducted to a secondary side of the second heat exchanger, preferably to a secondary side of the second heat exchanger and to a secondary side of the third heat exchanger, most preferably to a secondary side of a first section of the second heat exchanger, to a secondary side of a second section of the second heat exchanger and to a secondary side of the third heat exchanger.
8. An apparatus for use by the method according to any one of the preceding claims and comprising a boiler and a heat exchanger, characterised in that the apparatus further comprises

- a column provided with an inlet for a liquid containing more volatile and less volatile components, a discharge for more volatile components, and an outlet for less volatile components,

5     - a first heat exchanger provided with an inlet and an outlet of a primary side for the more volatile components and an inlet and an outlet of a secondary side for the less volatile components,

- a separator with an inlet, which is connected with the outlet from the primary side for the more volatile components.

10     9. An apparatus according to claim 8, characterised in that the apparatus further comprises a compressor, which is arranged between the outlet in the column for the more volatile components and the inlet of the first heat exchanger for the more volatile components.

15     10. An apparatus according to claim 8 or 9, characterised in that it further comprises

20     - a second heat exchanger having an inlet to a primary side from the outlet of the primary side of the first heat exchanger, an outlet from the primary side, an inlet to a secondary side for the liquid containing the more volatile components and the less volatile components, and an outlet from the secondary side for the liquid containing the more volatile components and the less volatile components, wherein the outlet from the primary side is in connection with the inlet to the separator, and

25     - a third heat exchanger with an inlet to a primary side for a gaseous fraction of the more volatile components from an outlet from the separator and an outlet from the primary side for a condensate of the more volatile components.



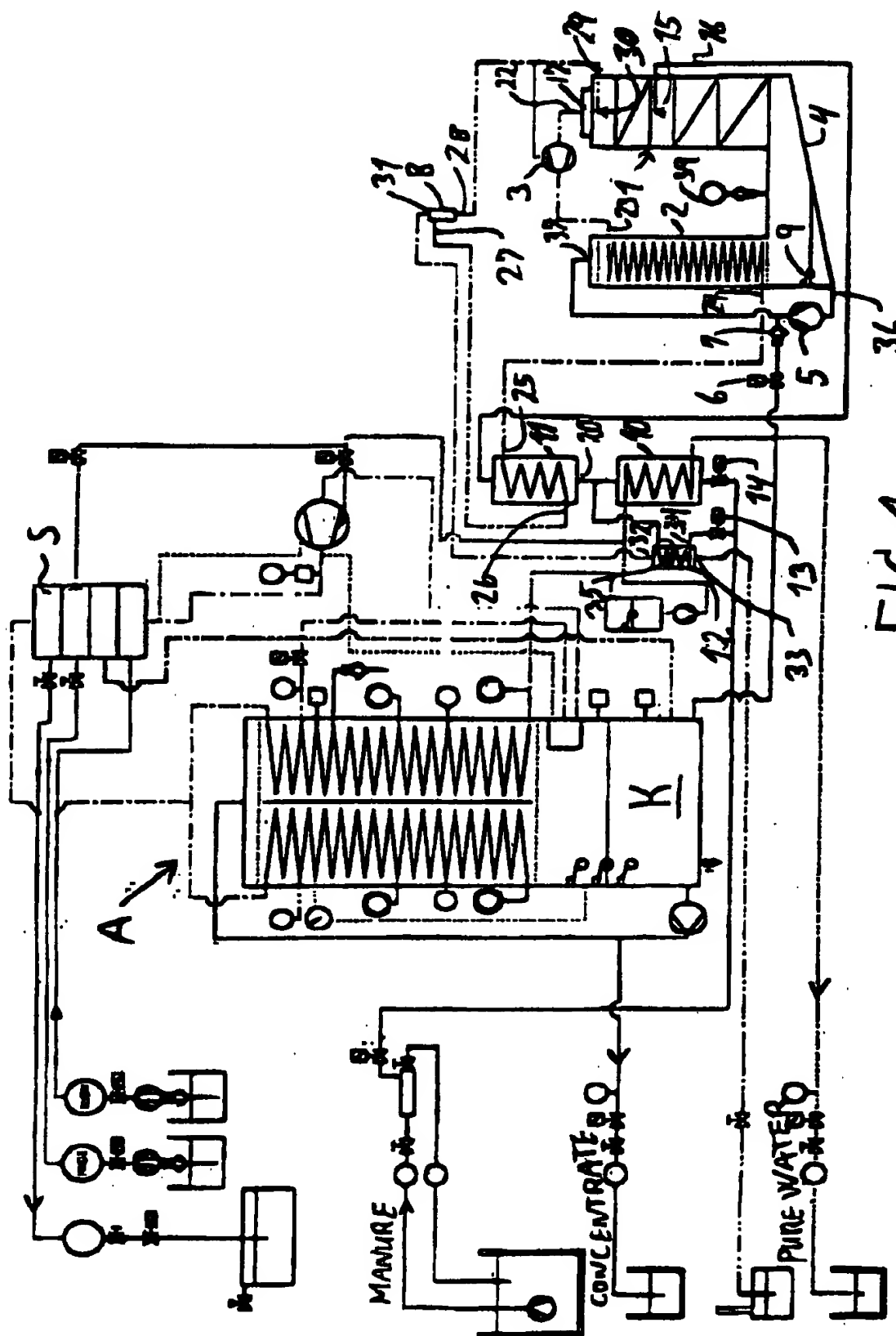


FIG. 1

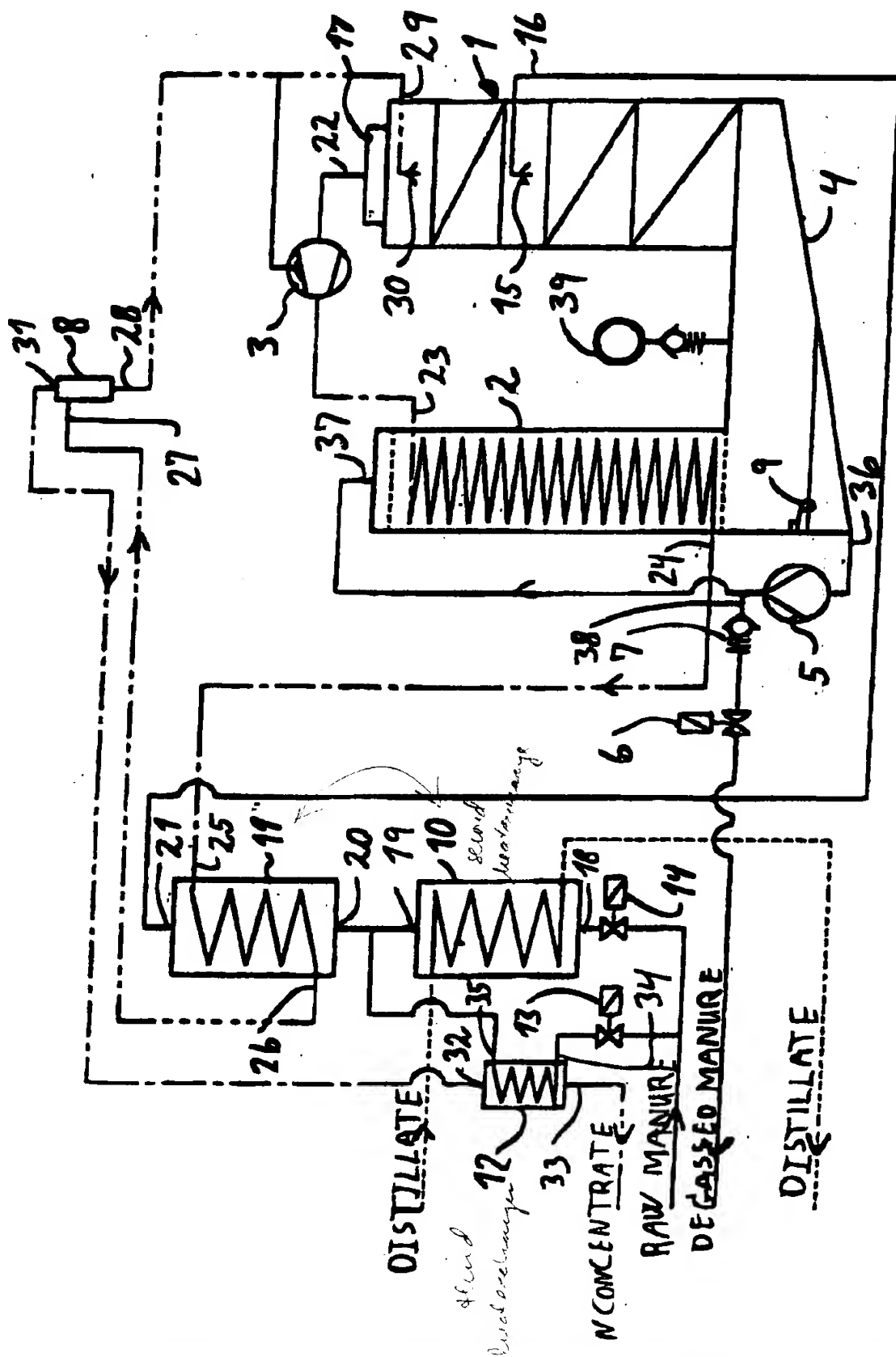


FIG. 2

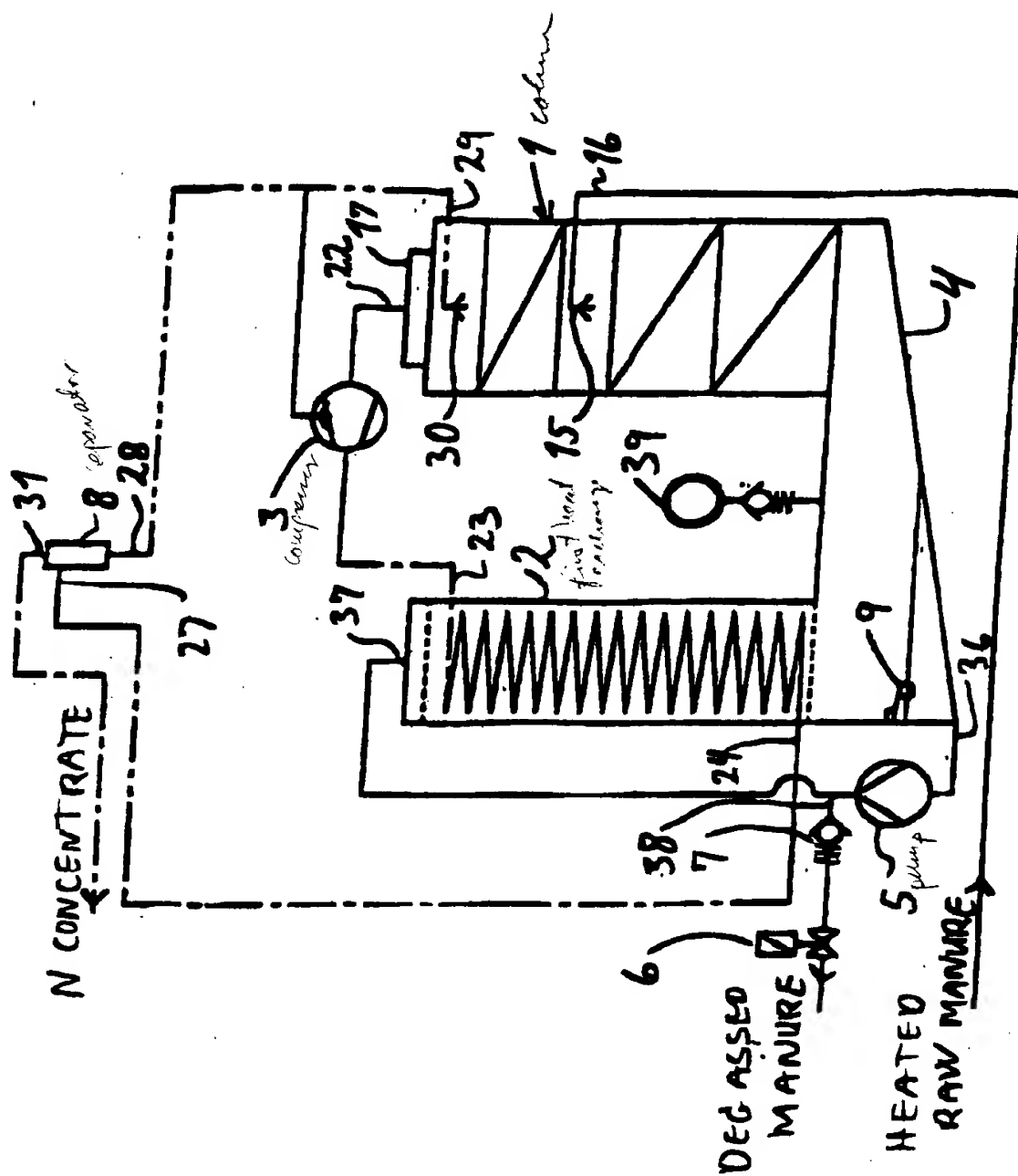


FIG. 3

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/DK 97/00521

## A. CLASSIFICATION OF SUBJECT MATTER

IPC6: C02F 1/20, C02F 1/04, B01D 19/00, B01D 1/00, F25J 3/02  
According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: B01D, C02F, F25J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

QUESTEL: WPIL

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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☐ Further documents are listed in the continuation of Box C.

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